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ULTRASONIC PROCESS FOR CURING ADHESIVES

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The objective of this program was to demonstrate in the laboratory the feasibility of using ultrasonic energy to cure a structural adhesive used to bond two pieces of 2024-T3 aluminum. A process was developed that demonstrated that American Cyanamid FM-73 adhesive could be cured using ultrasonic energy at a power level of less than 25 watts/sq. inch of adhesive area. The bonds produced using ultrasonic energy were just as strong as the bonds produced using a thermal process. A styrene butadiene rubber was found to be an excellent material to couple the ultrasonic energy to the adhesive through the aluminum substrate. The required coupling pressure ranged from 10 to 15 psi. Ultrasonic energy is absorbed by the adhesive and is converted to heat. Fine thermocouples embedded in the adhesive showed that the temperature of the adhesive increased from room temperature (70°F) to 250°F in less than 10 minutes.

(Continued on attached sheet)

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13. ABSTRACT (Concluded)

The method currently used to cure the epoxy adhesive bonding a composite repair patch to an aircraft structure employs a heating blanket over the patch and the surrounding aircraft structure. This method is inefficient and requires the area surrounding a patch to be heated to the same temperature required to cure the adhesive. Since ultrasonic energy is absorbed directly by the adhesive, very little energy is wasted heating up the area surrounding the patch. Thus, an ultrasonic heating process is more efficient than the heating blanket method. The ultrasonic process has the potential to be used for repairing aging aircraft structures with bonded composite patches.

PREFACE

This report covers Battelle's work performed from June 1992 to February 1993 under Subcontract No. RI-78529X from The University of Dayton Research Institute under Air Force Contract No. F33615-89-C-5643. This project was administered under the direction of Mr. D. Robert Askins of The University of Dayton Research Institute.

The Principal Investigator on this project was Dr. Nagabhusan Senapati. He was assisted by Mr. Ronald Moulder on the ultrasonic process development, by Mr. Samuel Smith on structural assessment and testing and by Mr. Albert Bunk on adhesive curing.

This report was submitted by Battelle to the University of Dayton Research Institute on March 1, 1993.

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FIGURES

Introduction

There is a growing need in the United States Air Force to repair and maintain its fleet of aging aircraft. In the conventional repair of aircraft structures, aluminum doublers are fastened to the structure to patch stress cracks or holes. However, the potential for fatigue cracking at fastener holes and inspectibility problems with conventional metal doubler repairs prompted consideration of bonded metal or composite repairs. A repair system using heat blankets to cure boron-epoxy patches has been successfully developed by the Australian Air Force. Bonded boron-epoxy repair patches were shown to provide excellent durability and damage tolerance characteristics as well as being inspectable using eddy current techniques.

In order to ensure adequate heating of the adhesive using the thermal blanket process, the entire assembly, i.e. the patch, adhesive and metal structure, must be heated and held at an elevated temperature for several hours. In addition there is some concern about the strength of the adhesive bonding the boron-epoxy patch to the structure. Therefore, a need exists to explore alternative means to heat the adhesive and to minimize heating of the metal structure as well as to improve the strength of the joint.

This report is on the technical feasibility of an "Ultrasonic Process for Curing FM-73 Structural Adhesive".

Objective

The specific objective of this laboratory study was to evaluate the feasibility of using ultrasonic energy to cure FM-73 structural adhesive to bond coupon size 2024-T3 bare aluminum alloy phosphoric acid anodized and primed with BR-127 primer.

Technical Approach

Ultrasonic energy is a form of mechanical stress waves which propagate through a medium. The transmission and the absorption of the ultrasonic energy in the medium depends on the specific acoustic impedance and the absorption coefficient of the medium. Uncured epoxy type adhesives are known to have very high absorption coefficients and therefore are expected to absorb ultrasonic energy and convert the energy to heat. The poor thermal conductivity of typical epoxy type adhesives

becomes an advantage in retaining the heat in the adhesive and reducing the temperature of the aluminum substrate. Ultrasonic energy is also known to accelerate chemical reactions, increase the rate of cure, and also improve the wetting of the adhesive with the substrate due to shear thinning.

One of the key steps to demonstrate the feasibility of using ultrasonic energy to cure structural adhesive like FM-73 is to identify a suitable coupling medium to transmit ultrasonic energy from the source, (e.g., the vibrating face of an ultrasonic horn) to the adhesive. The other key process variables are the ultrasonic intensity, the exposure time, and the coupling pressure. A statistically designed matrix of experiments was planned to evaluate the process variables and to determine the preferred conditions for curing the FM-73 adhesive. The degree of cure was initially evaluated visually. The state of cure was further evaluated on selected specimens by using a differential scanning calorimeter (DSC). The bond strength was also evaluated by wedge and lap shear tests.

Experimental Procedure

Figure 1 is a schematic of the laboratory test fixture used in this program. The test fixture consisted of an ultrasonic transducer, booster, horn or waveguide, specimen holder, load cell, and hydraulic cylinder used to apply pressure on the test specimen. The following instruments were used.

- Branson Model 105 transducer rated for 1000 watts
- Branson 1:2.5 booster
- Rectangular horn (1.5 x 3 inches)
- ENI Model EGR 800 ultrasonic generator/amplifier
- Data Precision Model 5845 frequency counter
- Type K thermocouple (0.002 inch diameter)
- Doric Model 410A thermocouple readout
- A 0 to 200 pound load cell with a readout meter
- Strip chart recorder used to record the temperature of the adhesive.

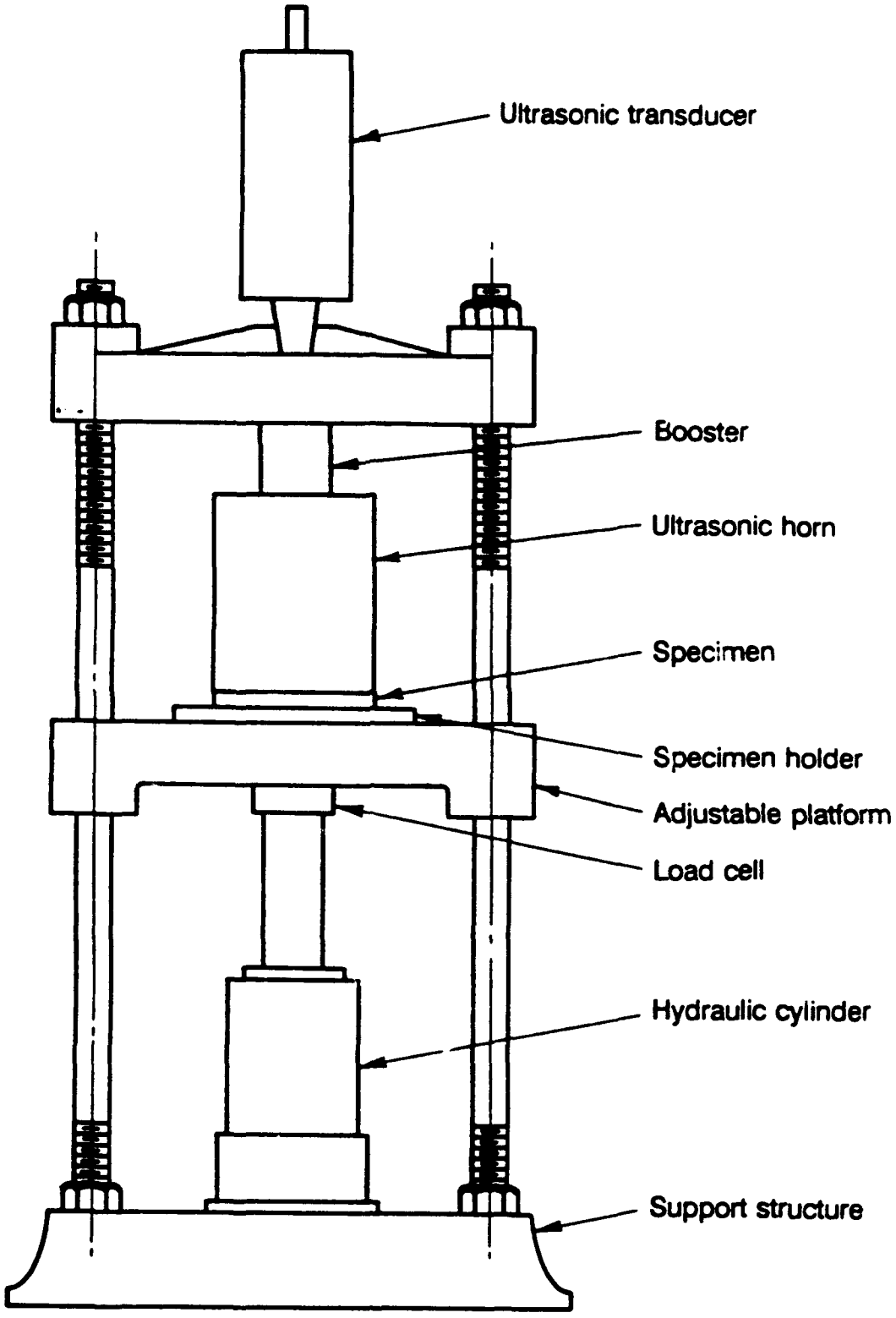

Figure 1. Schematic experimental setup for ultrasonic activation of adhesives.

The aluminum test coupons were cut from 0.125-inch-thick 2024-T3 bare aluminum. For the coupling tests 1 x 6 inch aluminum coupons were used. For the parametric tests 1 x 4 inch aluminum coupons were used. These coupons were phosphoric acid anodized (PAA) and primed with BR-127 primer. Anodizing of the aluminum was done in accordance with Boeing Aircraft Company Specification BAC 5555. The sheet aluminum was cut to the test coupon size (1 x 4 inches). The aluminum coupons were then cleaned using MEK solvent per Boeing specification BAC 5750. After being cleaned the coupons were wrapped in brown Kraft paper until they could be alkaline cleaned per Boeing Specification BAC 5514 and BAC 5749 Method 1, soak cleaning using Turco 4215 and 4215 additive. The coupons were deoxidized per BAC 5514 using P.2 etch with ferric sulfate and sulfuric acid in deionized water at 150±5°F. After cold water rinsing per BAC 5555 the coupons were anodized and dried per BAC 5555. The anodized coupons were checked using white or near-white light per BAC 5555. After anodizing the coupons were wrapped in Kraft paper for storage.

The anodized aluminum coupons were primed using American Cyanamid BR-127 primer per the manufacturer's instructions. The primer was applied to a thickness of 0.0001 to 0.0003 inch. The thickness was determined by matching the color of the primed panels with the standard color panels supplied by the manufacturer. A thickness gage was also used to spot check some panels.

For all tests, American Cyanamid FM-73 adhesive with a polyester knit was used. The adhesive was 0.010-inch thick and was tacky on both sides. The adhesive was stored at 0°F and allowed to come to room temperature in a sealed bag before it was used. For all tests a single layer of 1 x 4 inch strip of adhesive was used.

The adhesive was placed between two pieces of aluminum and the specimen was then placed in the holder on the test fixture. The specimen holder was designed to isolate the specimen acoustically and thermally and to prevent the test specimen from moving during the experiment. After the test specimen was placed in the test fixture, the hydraulic cylinder was activated until the desired pressure was applied on the test specimen. The pressure was determined by dividing the force on the test specimen by the area of the adhesive. Once the pressure was applied, power to the ultrasonic transducer was turned on for the desired time. Following ultrasonic exposure the pressure was maintained for an additional 5 minutes before slowly being released. The nominal operating frequency of the ultrasonic system was 20,000 Hz.

In order to check on the anodizing and priming procedures used and to serve as control, several thermally cured specimens were prepared. These specimens were tested for strength using ASTM test standard D-3762, Test Method for Adhesive-Bonded Surface Durability of Aluminum

(wedge test) and ASTM test standard D-3167, Test Method for Floating Roller Peel Resistance on Adhesives (peel test). These thermally cured specimens were cured in a hot press under a pressure of 30 psi. The temperature in the hot press was increased at a rate of 5°F per minute. After exposure to a temperature of 250°F for 60 minutes the specimen was allowed to cool down in the press to 150°F before they were removed from the press. Two series of wedge and peel tests were conducted. A series of tests was done each time a batch of aluminum coupons were anodized and primed.

The results of the wedge and peel tests were as follows:

- First series of tests

  Peel test: 47.94 lbs/in. (average) after a 10 minute soak at -67°F
  Wedge test: Initial crack length — 0.94 inch
  Crack growth after 1 hour — 0.02 inch
  Crack growth after 4 hours — 0.0
  Crack growth after 24 hours — 0.02 inch
  All failures were cohesive.
  Specimens were tested at 140°F and 96 percent relative humidity.

- Second series of tests

  Peel test: 45.92 lbs/in. (average) after a 10 minute soak at -67°F
  Wedge test: Initial crack length — 1.05 inches
  Crack growth after 1 hour — 0.01 inch
  Crack growth after 4 hours — 0.01 inch
  Crack growth after 24 hours — 0.02 inch.
  All failures were cohesive.
  Specimens were tested at 140°F and 96 percent relative humidity.

Based on the above results it was concluded that the anodizing and priming techniques used in our laboratories were excellent.

Test Results

Coupling Tests

For most applications where ultrasonic energy is used, a coupling material is required between the ultrasonic horn and test specimen. Several different couplants where investigated under this program. The couplants were:

1. No couplant or direct contact
2. Polyisobutadiene (PIB)
3. Natural rubber
4. Silicone rubber
5. Silicone RTV
6. Echo Ultrasound grade 60 Thermosonic
7. Echo Ultrasound grade 100 Pyrogel
8. Echo Ultrasound Sonotemp
9. Teflon tape
10. Red styrene butadiene rubber (RR).

The benefit of each couplant was determined by visual inspection of the couplant and top surface of the aluminum test specimen and the time-temperature curve of the adhesive. The couplants were tested using a power range of 100 to 300 watts and a pressure range of 12 to 15 psi.

With no couplant, the coupling was very poor and the adhesive was under cured. Also, the top surface of the aluminum was damaged slightly. Except for the PIB and red rubber (RR), the other couplants dissipated due to the heat of the test specimen and the shear thinning effect of the ultrasonic energy. At high power settings (i.e., above 150 watts) the red rubber tended to burn. In addition time-temperature data showed that the red rubber was not as efficient as PIB in coupling energy into the test specimen. For these two reasons it was initially decided to use PIB to couple ultrasonic

energy into the test specimen. One of the disadvantages of using of PIB is that it can only be cleaned up using a volatile solvent such as acetone.

During the coupling tests it was determined that too much ultrasonic energy and heat was passing from the specimen into the test fixture. To correct this problem a new specimen holder was built and strips (1/16 inch thick by 1/4 inch wide) of cellulose were placed under the test specimen to isolate the specimen from the specimen holder. Figure 2 shows the time-temperature curves at 100 watts using PIB or red rubber. This figure shows that the temperature of the adhesive reaches a maximum in the first 10 minutes using PIB faster than it does using red rubber. All time-temperature graphs were normalized to a starting temperature of 70°F.

Parametric Tests

Based on the results of the coupling tests the following test variable parameters were selected for the first series of tests (Box-Behnken tests):

Power (W):	50, 75, and 150 watts
Pressure (P):	5, 10, and 15 psi
Time (T):	5, 30, and 60 minutes.

In the Box-Behnken experimental design the following conditions were evaluated:

$W_{50}\ T_5\ P_5$, $W_{50}\ T_5\ P_{10}$, $W_{50}\ T_{30}\ P_5$, $W_{50}\ T_{30}\ P_{15}$
$W_{50}\ T_{60}\ P_{10}$, $W_{75}\ T_5\ P_5$, $W_{75}\ T_5\ P_{15}$, $W_{75}\ T_{30}\ P_{10}$
$W_{75}\ T_{60}\ P_5$, $W_{75}\ T_{60}\ P_{15}$, $W_{150}\ T_5\ P_{10}$, $W_{150}\ T_{30}\ P_5$
$W_{150}\ T_{30}\ P_{15}$, $W_{150}\ T_{60}\ P_{10}$, and $W_{150}\ T_{60}\ P_{15}$.

There are a total of 15 combinations. The two end points ($W_{50}\ T_5\ P_5$ and $W_{150}\ T_{60}\ P_{15}$) were added to the Box-Behnken experimental design. A total of five replicates were done for each combination. The tests were done in random order and an additional five tests were conducted for the center combination ($W_{75}\ T_{30}\ P_{10}$).

After exposure to ultrasonic energy, the test specimens were taken apart using a chisel. The degree of cure was determined visually by the color of the adhesive, its tackiness, and the amount of effort required to pry apart the test specimen. A thermocouple was used in some specimens to monitor the temperature of the adhesive. An attempt was made to determine the degree of adhesive

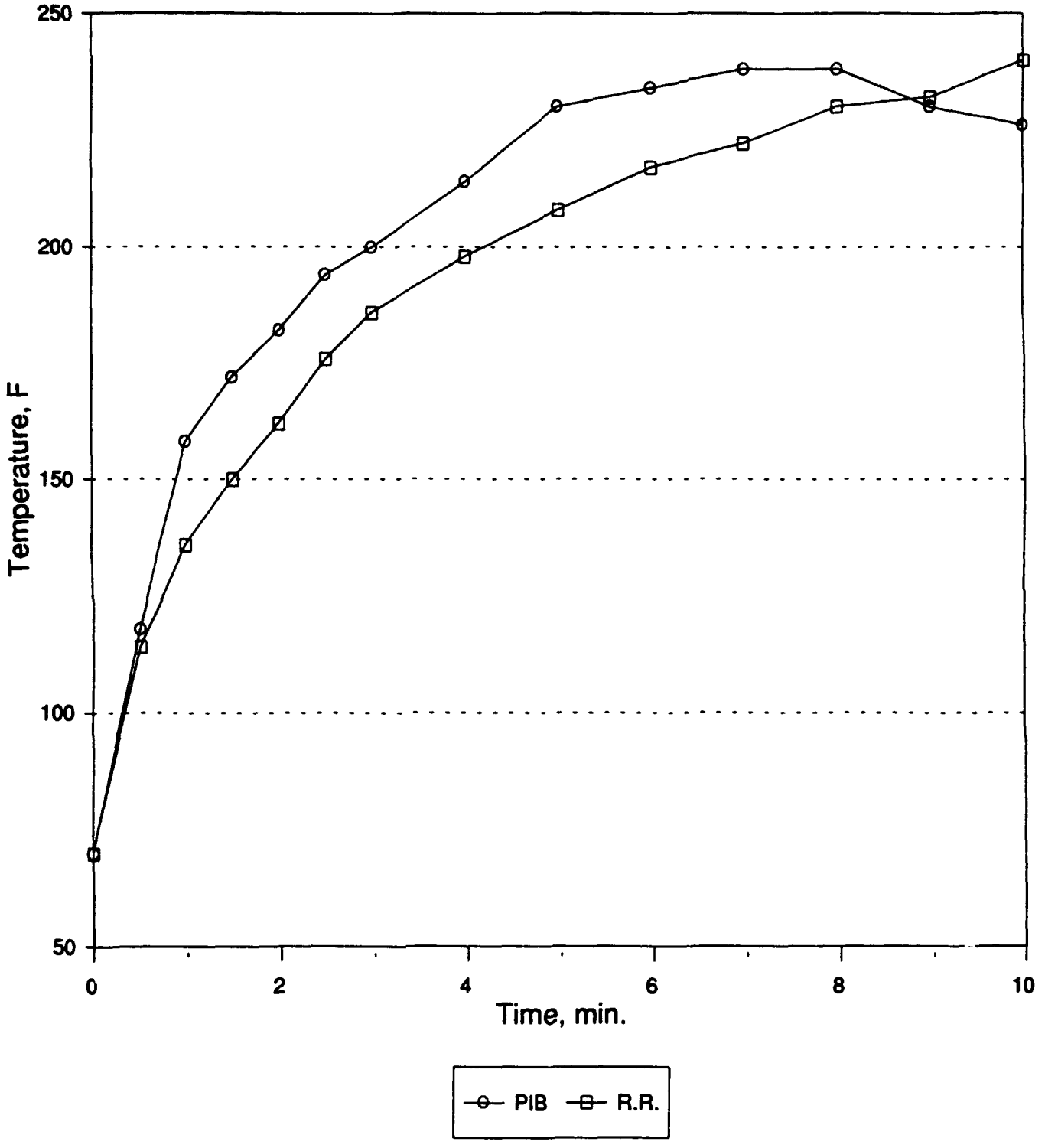

Figure 2. Comparison of the adhesive temperature using PIB or RR at 100 watts.

cure by measuring its hardness using a Shore A hardness gage or scratch test. Both test procedures were unsatisfactory because the adhesive was too thin to obtain good readings. DSC tests were conducted on six specimens from the first series of tests.

The DSC results were as follows:

1. Specimen Number 100609 (50 watts, 60 minutes, and 10 psi)
   Good cure

2. Specimen Number 92804 (75 watts, 5 minutes, and 5 psi)
   Under Cured

3. Specimen Number 100601 (75 watts, 30 minutes, and 10 psi)
   Good cure, as good a cure as for the thermally cure specimen

4. Specimen Number 100605 (75 watts, 30 minutes, and 10 psi)
   Same results as for Specimen Number 100601

5. Specimen Number 100106 (75 watts, 60 minutes, and 5 psi)
   Best cure of all specimens including thermally cured specimen

6. Specimen Number 100209 (150 watts, 5 minutes, and 10 psi)
   Not cured properly, rapid heating may have damaged adhesive cross links

7. Thermally cured specimen (60 minutes at 250°F)
   Good cure.

Copies of the DSC test curves are in the appendix of this report. Figures 3 and 4 are plots of the temperature of the adhesive as a functional of time for several different test conditions. These graphs show that at 150 watts the temperature of the adhesive exceeded 350°F. This is too high a temperature to cure the adhesive properly.

Based on visual observations, the time-temperature curves, and the DSC test results, the following test conditions were selected for the second series of tests:

60 watts for 45, 60, 75, and 90 minutes
75 watts for 30, 45, and 75 minutes
90 watts for 30 and 45 minutes.

For the second series of tests it was decided to switch to a larger horn (1¼ x 8¼ inches) since a larger horn might be required to cure the larger wedge test specimens necessary to demonstrate the adhesive bond strengths achieved using ultrasonic energy.

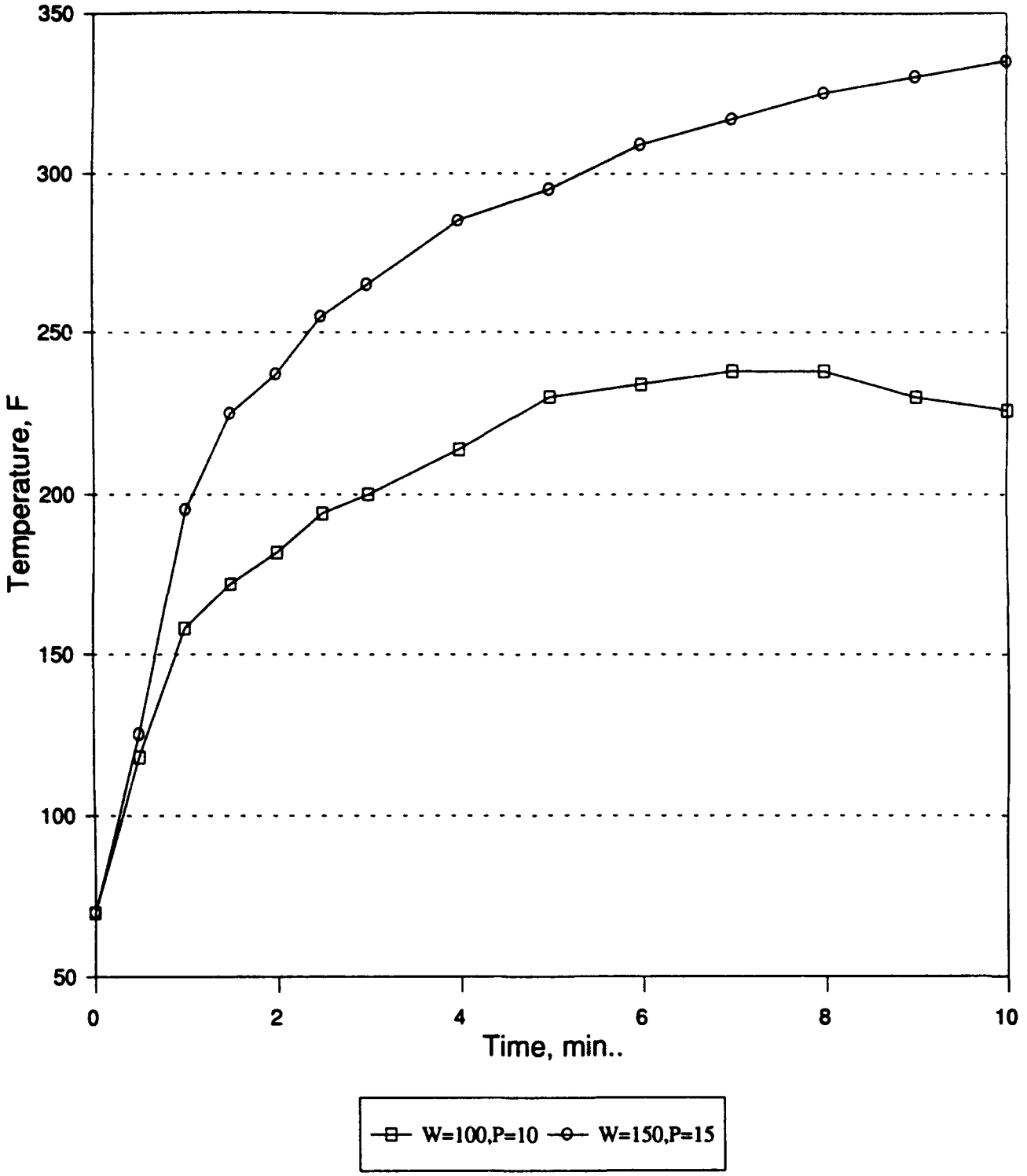

Figure 3. Comparison of the temperature of the adhesive for two different power settings using PIB couplant.

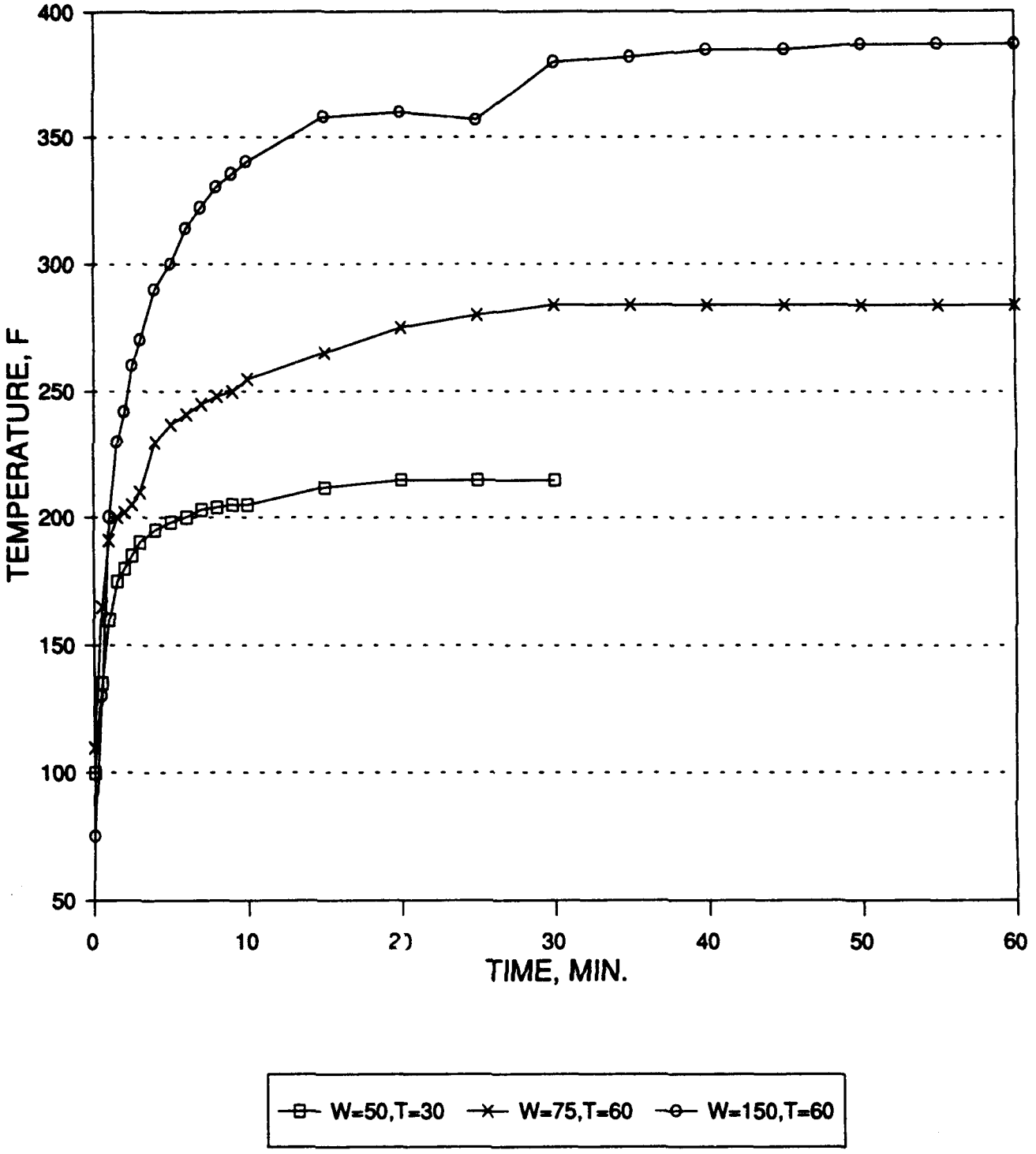

Figure 4. **Comparison of the temperature of the adhesive for three different power settings using PIB couplant.**

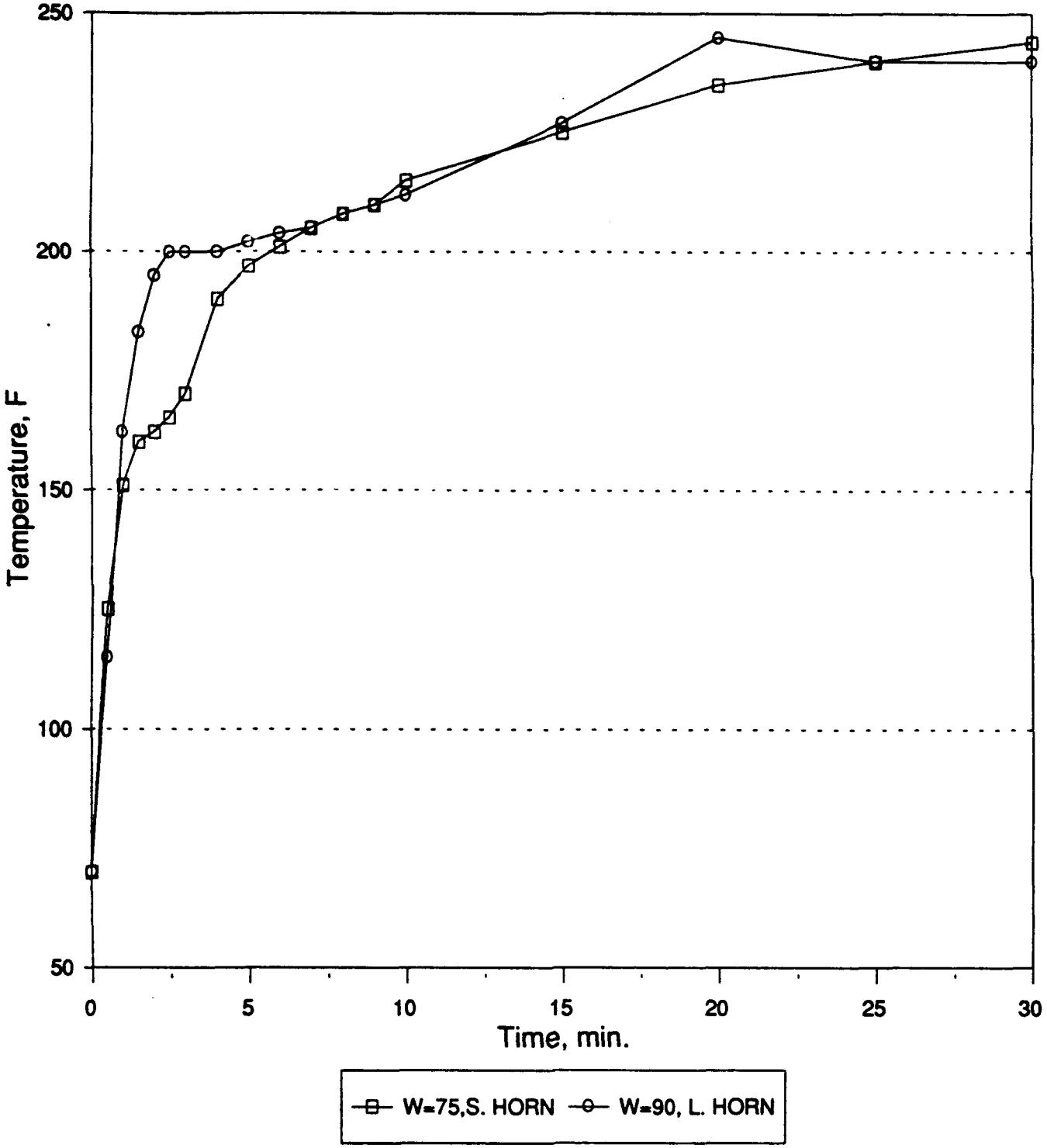

Figure 5. Comparison of adhesive temperature using a large horn and a small horn (PIB couplant).

Preliminary tests with the large horn produced specimens with the adhesive under cured. The same test parameters that produced good results with the small horn were used with the large horn. Because of the greater heat sink effect of the large horn compared with the small horn, we believed that the adhesive was not reaching a high enough temperature to cure properly. When the power to the transducer was increased from 75 to 90 watts, the time-temperature curved showed similar results as for the 75 watt input for the smaller horn (see Figure 5). However, the adhesive strengths were poor when the large horn was used. The adhesive appeared to be over cured in the middle of the 1 x 6 inch test specimen. We have not been able to determine why the adhesive in the middle of the test specimen was over cured resulting in weak bond strengths.

In order to produce better test specimens, the red rubber material was again tried as the couplant material. The use of this material produced a more uniform adhesive cure. It was then decided to use red rubber as the couplant material for the wedge and the lap shear test specimens. Also the small horn was used to prepare these specimens. In order to save time and project funds it was decided with approval from the Air Force that the second series of tests would not be conducted. The final bonding combinations were used to produce the wedge and lap shear test specimens. The following test combinations were used to produce the wedge and lap shear test specimens:

65 watts for 60 minutes
75 watts for 30 and 60 minutes
85 watts for 60 minutes
10 psi pressure for all tests.

The wedge test specimens were 1 x 6 inches with a 1 x 5 inch adhesive strip. The 3-inch-long horn was centered over the adhesive strip. Three specimens were produced for each test condition. The following wedge test results were obtained:

65 watts for 60 minutes

		Crack growth, inch		
	Initial, inch	1 hour	4 hours	24 hours
Specimen A:	0.96	0.02	0.04	0.04
Specimen B:	1.06	0.05	0	0.01
Specimen C:	0.97	0.06	0	0.01

75 watts for 30 minutes

	Initial, inch	Crack growth, inch 1 hour	4 hours	24 hours
Specimen A:	1.00	0.16	0	0.04
Specimen B:	0.95	0.10	0.02	0
Specimen C:	0.92	0.06	0.04	0

75 watts for 60 minutes

	Initial, inch	Crack growth, inch 1 hour	4 hours	24 hours
Specimen A:	1.13	0.02	0	0.01
Specimen B:	1.16	0.02	0	0.12
Specimen C:	0.92	0.12	0.02	0

85 watts for 60 minutes

	Initial, inch	Crack growth, inch 1 hour	4 hours	24 hours
Specimen A:	0.93	0.01	0	0.08
Specimen B:	0.86	0.25	0.04	0
Specimen C:	0.97	0	0.02	0

All wedge test specimens were conditioned and tested at 140°F and 96 percent relative humidity.

The failure mode for all of the specimens was cohesive. For comparison a thermally cured specimen nominally has the following values:

	Crack Growth, inch		
Initial, inch	**1 hour**	**4 hours**	**24 hours**
<1.30	≤ 0.05	≤ 0.05	≤ 0.05

To get an assessment of the state of the cure of the wedge test specimens under different ultrasonic cure conditions, DSC tests were conducted on two specimens for each of the four test conditions. The specimen numbers and curing conditions were as follows:

Specimen No.	Test Condition
11901	65 watts, 60 minutes
11702	65 watts, 60 minutes
11706	75 watts, 30 minutes
11703	75 watts, 30 minutes
11705	75 watts, 60 minutes
11701	75 watts, 60 minutes

11902	85 watts, 60 minutes
11704	85 watts, 60 minutes.

The DSC tests were conducted on small adhesive samples taken from these eight specimens. Figures 6 through 13 show the DSC plots for these tests. The solid line shows the initial heat flow rate when the specimens were heated at a rate of 10.0°C per minute. Evidence of an isotherm is shown by the "dip" in the solid line, which is an indication of the presence of uncured adhesive. A fully cured adhesive shows negliable evidence of an isotherm during initial heating. The dotted line shows the heat flow rate during the second scan after the specimen was quickly quenched and reheated at the same rate of 10.0°C per minute. During the second scan the specimen is fully cured. Therefore, a comparison of the first and the second scan is also a good indicator of the degree of cure.

A thorough analysis of the DSC plots is beyond the scope of this project. However from these eight DSC plots it appears as though the adhesive cured at 85 watts for 60 minutes is almost fully cured. Other specimens have some degree of partial cure. The degree of cure for the other specimens is high enough such that the wedge tests indicate low crack growth.

Lap shear test specimens were also prepared using the same bonding conditions used for the wedge test specimens. Figure 14 is a drawing of a lap shear test specimen. Three lap shear specimens for the same condition were done simultaneously. Three thermally cured specimens were also prepared for comparison purposes. All lap shear tests were conducted at 160°F temperature after 10 minutes soak per ASTM D-1002. The average shear tensile strength of the thermally cured specimens was 3,894 pounds per square inch. The tensile strengths ranged from 2,954 to 4,762 pounds per square inch. The failure mode was cohesive.

The shear tensile strengths of the ultrasonically cured specimens were as follows:

Specimen Curing Conditions	Shear Tensile Strength, psi
65 watts, 60 minutes	600
75 watts, 30 minutes	3
85 watts, 60 minutes	3,780
75 watts, 60 minutes	1,400

The above results are the average of three specimens for each test condition.

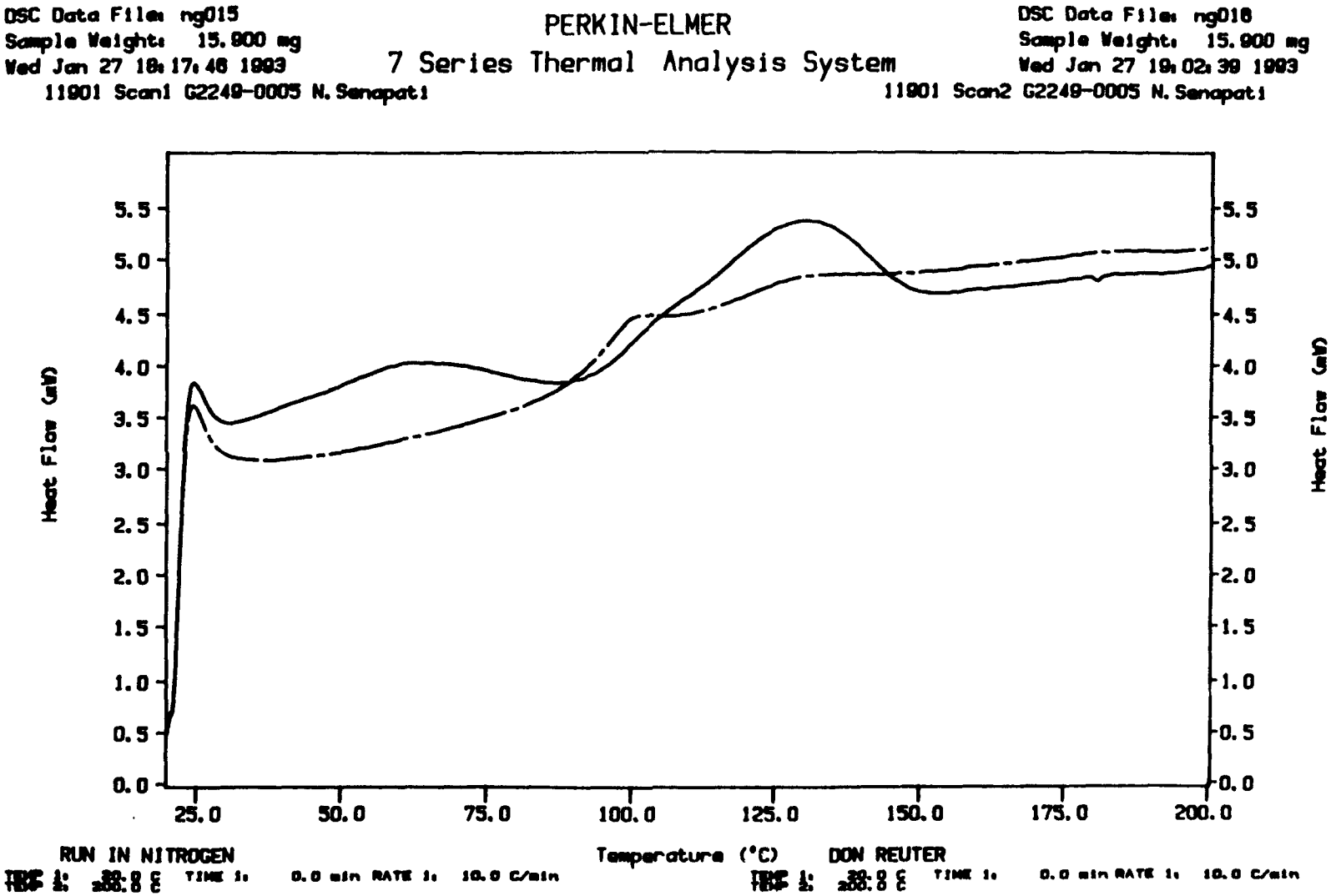

Figure 6. DSC plot of Specimen No. 11901.

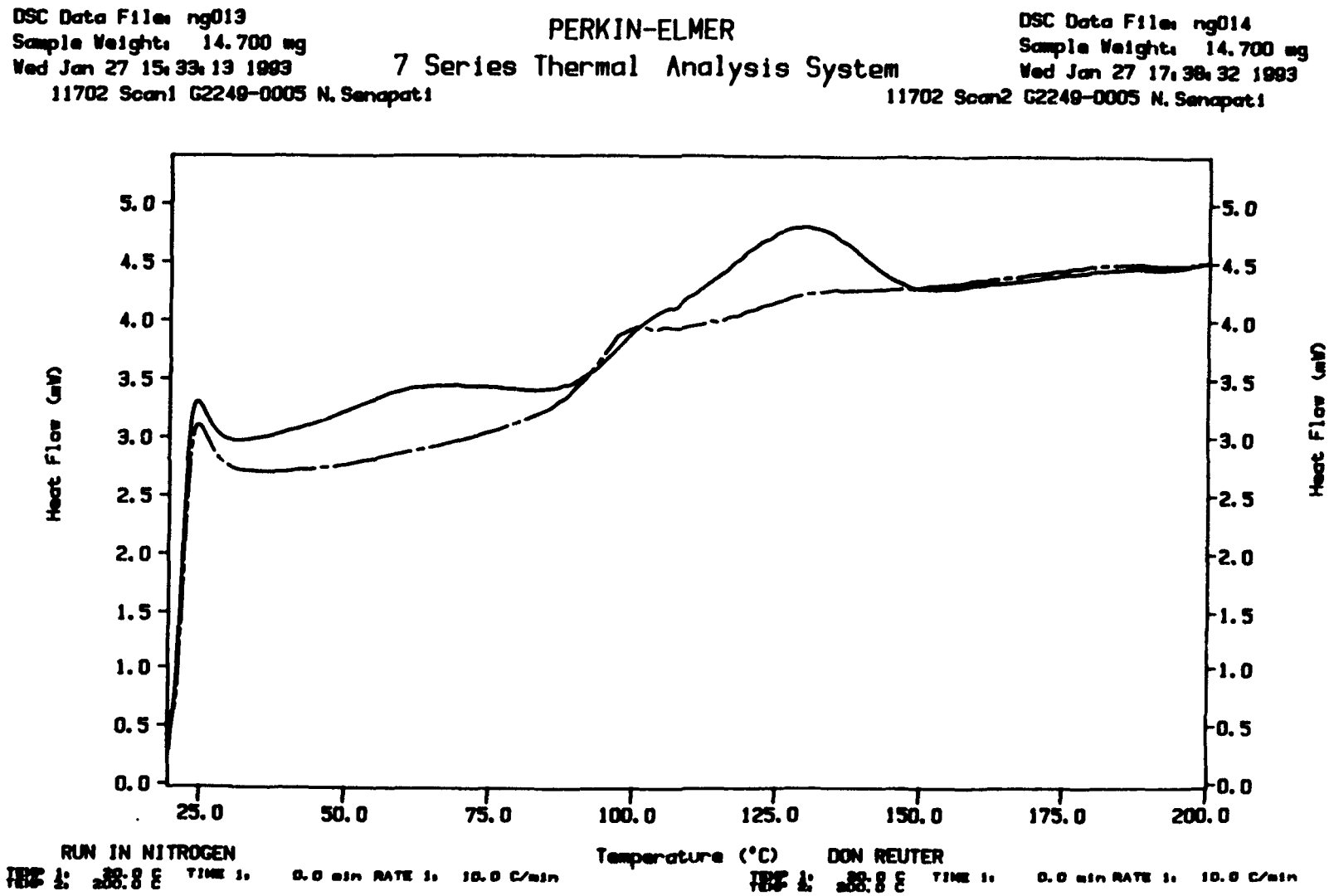

Figure 7. DSC plot of Specimen No. 11702.

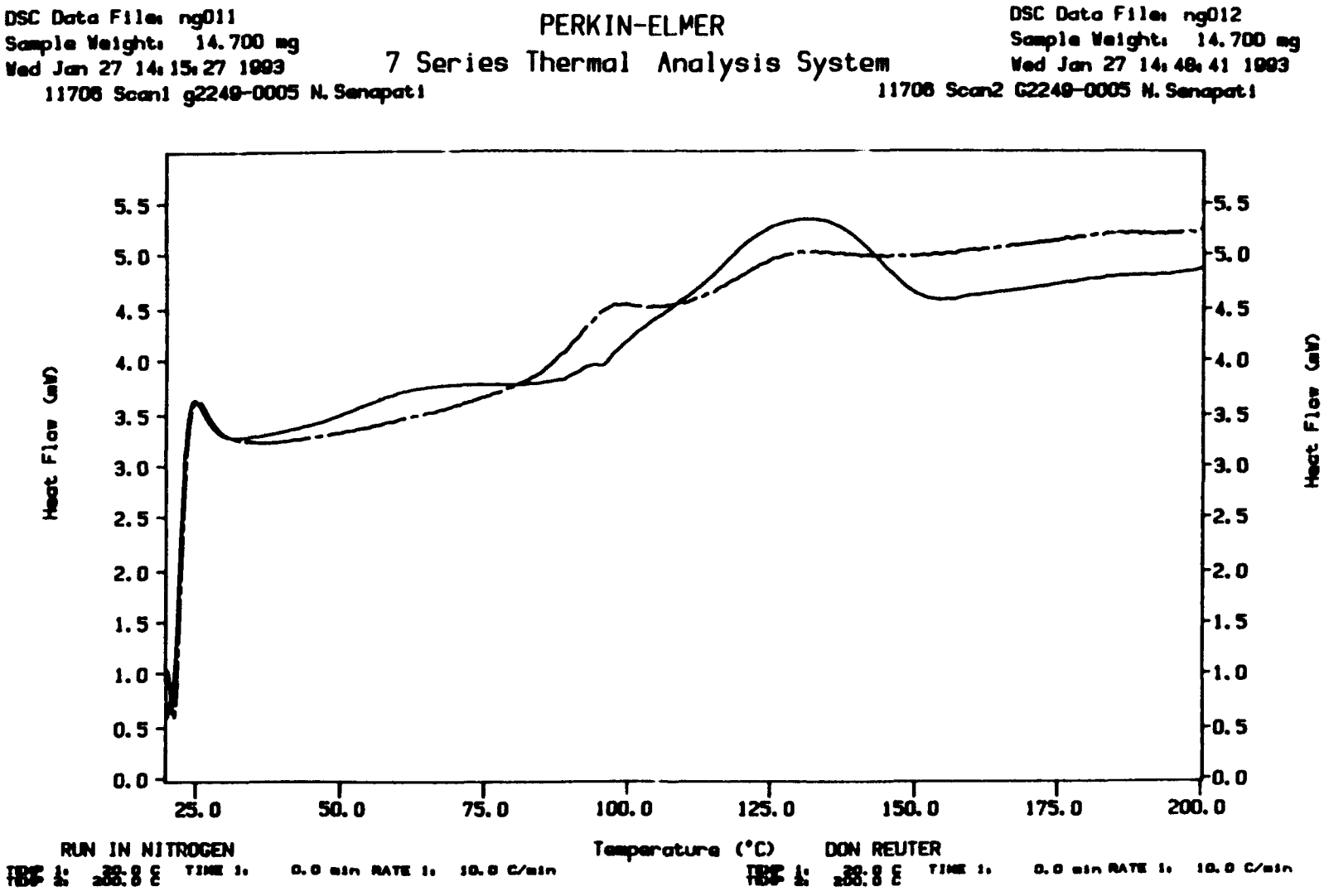

Figure 8. DSC plot of Specimen No. 11706.

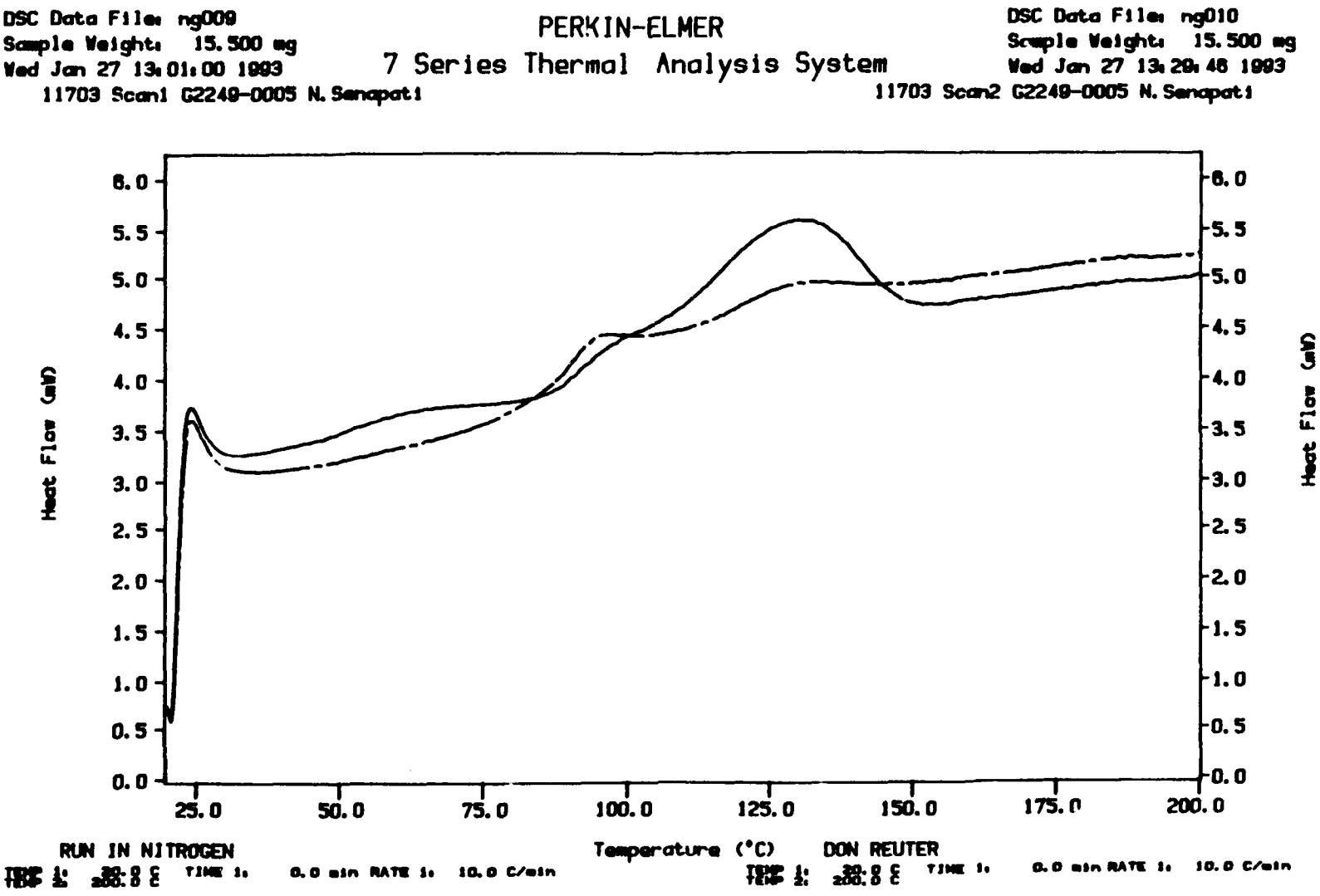

Figure 9. DSC plot of Specimen No. 11703.

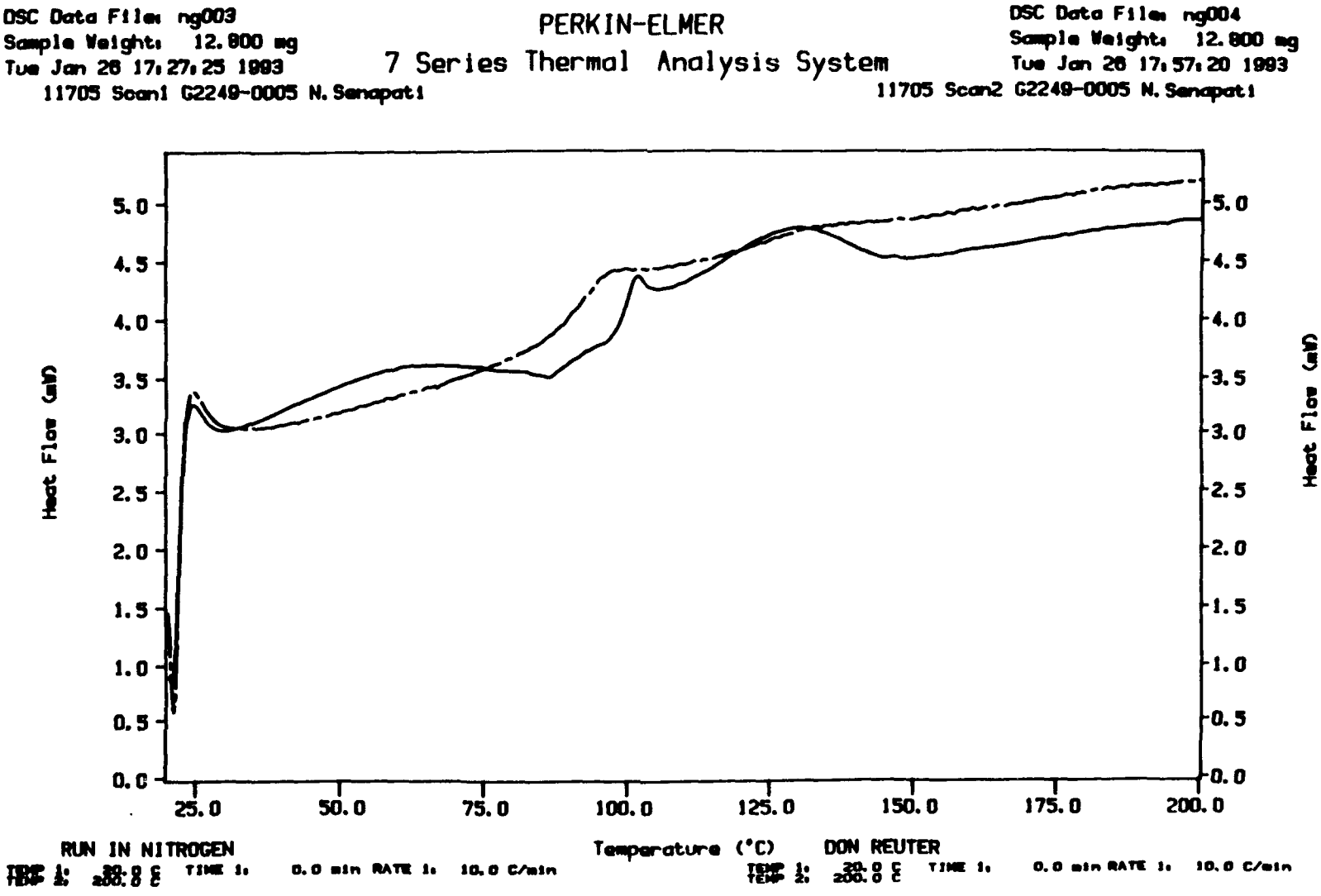

Figure 10. DSC plot of Specimen No. 11705.

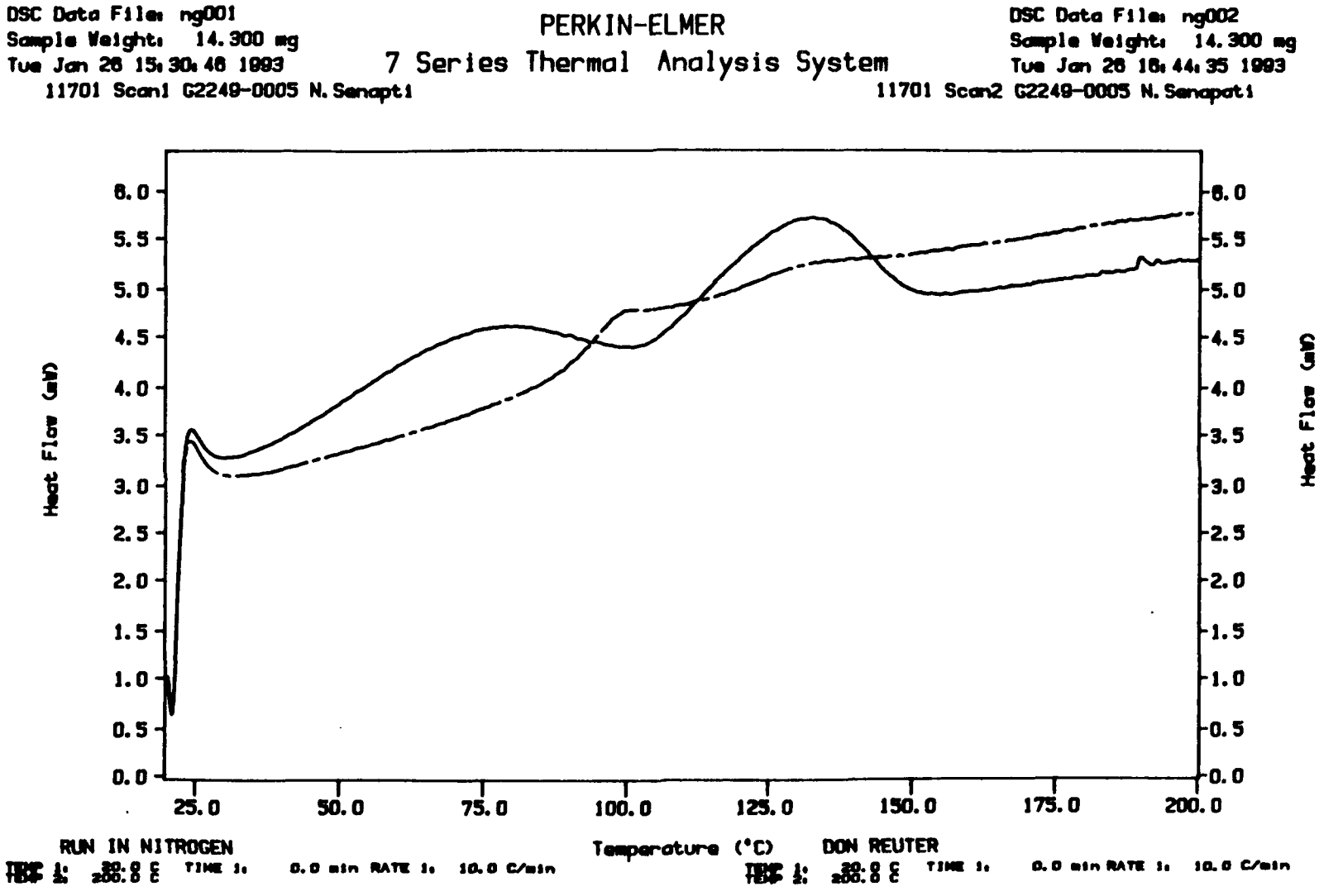

Figure 11. DSC plot of Specimen No. 11701.

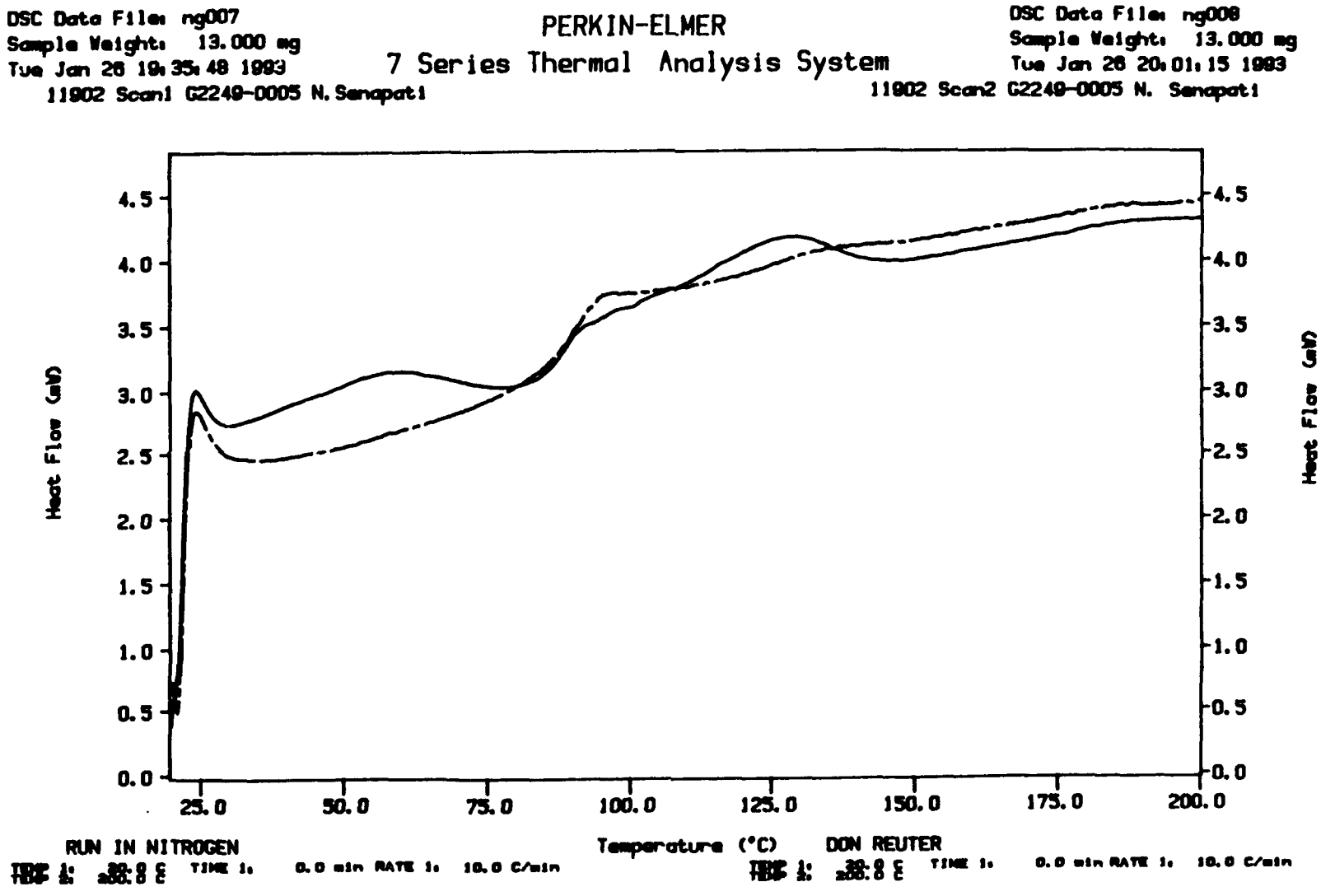

Figure 12. DSC plot of Specimen No. 11902.

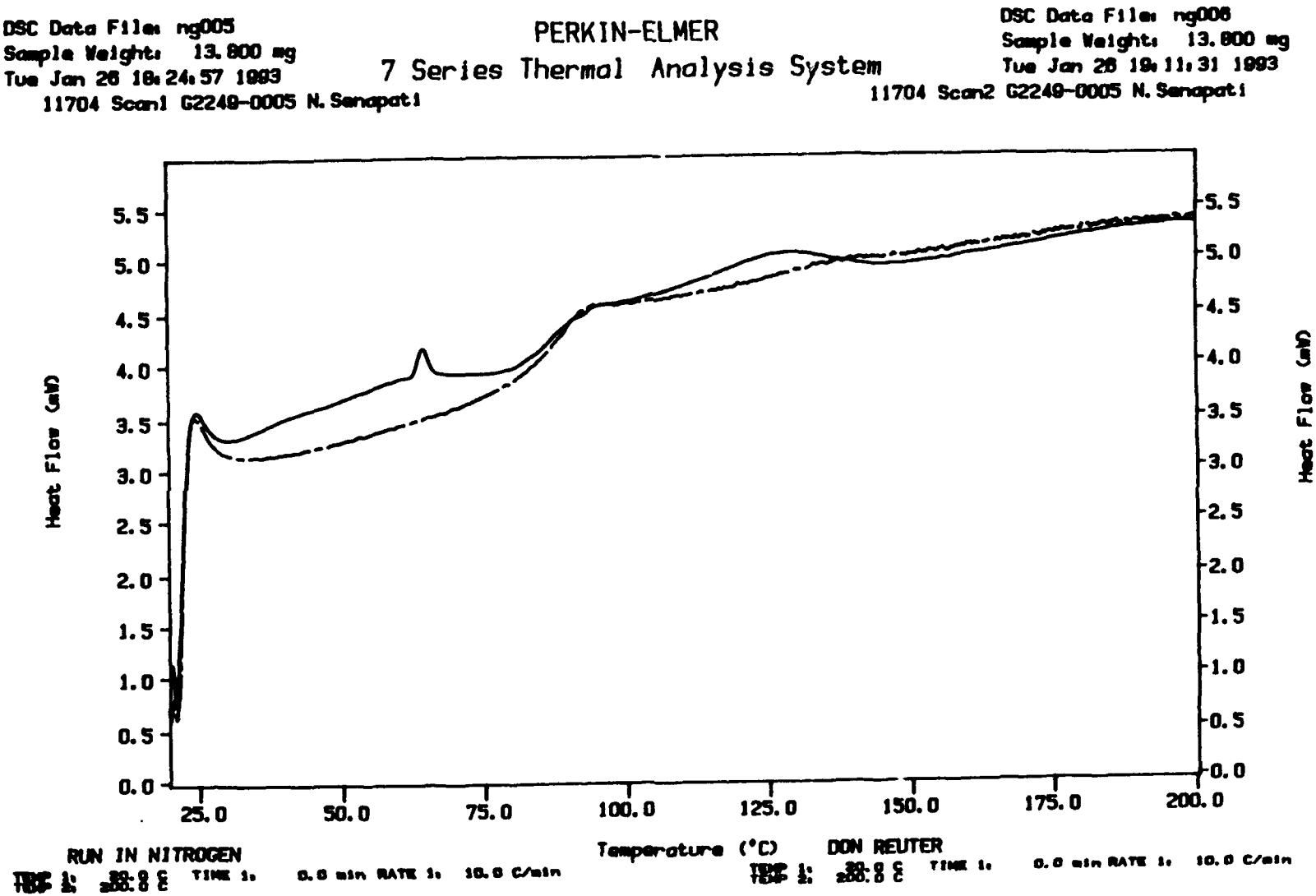

Figure 13. DSC plot of Specimen No. 11704.

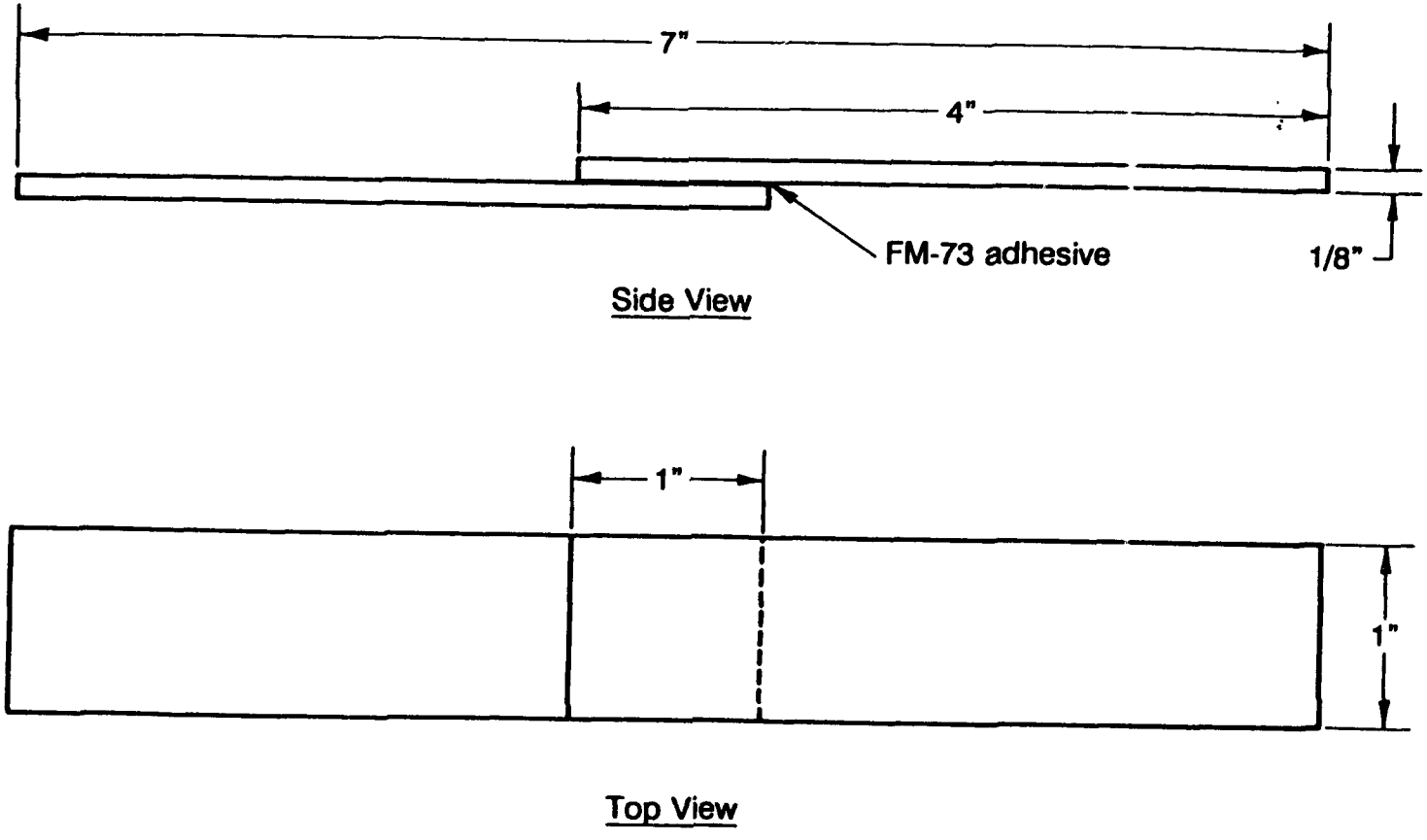

Figure 14. Lap shear aluminum test specimen with FM-73 adhesive.

The specimens cured at 65 and 75 watts were under cured and the failure mode was adhesion. For the test specimens cured at 85 watts the failure mode was cohesive. We believe the 65 and 75 watt specimens were under cured because of the heat sink effect of the aluminum. For the parametric tests a single 1 x 4 inch aluminum specimen was cured at a time. For the lap shear tests, three specimens at one time were exposed to ultrasonic energy. Thus, there was 3 times as much aluminum dissipating heat during the lap shear curing tests than there was for the parametric tests. Because of this greater heat dissipation, the lap shear specimens did not reach the required temperature. Aluminum is an excellent conductor of heat and the tests show that it is necessary to compensate for the heat sink effect of the aluminum to achieve a desired level of cure.

Conclusions

In this feasibility study we have successfully demonstrated that ultrasonic energy can be effectively used to cure structural adhesives like FM-73 to bond 2024-T3 aircraft aluminum. The ultrasonic intensity level necessary to achieve a full cure within 1 hour is less than 25 watts per square inch of adhesive or bond area. The ultrasonic energy is absorbed by the adhesive causing its temperature to increase very rapidly. Our observations indicate that the adhesive temperatures can be increased from room temperature (70°F) to 250°F in about 10 minutes.

Unlike the use of induction heat to cure adhesives, ultrasonic activation of FM-73 does not require the use of any additives in the adhesive. A styrene butadiene rubber was found to be an excellent coupling agent to couple the ultrasonic energy from the vibrating ultrasonic source to the aluminum structure and then into the adhesive. A coupling pressure between 10 and 15 psi was found to be more than adequate to couple the ultrasonic energy. At lower pressures the coupling was not as efficient. There is no need to increase the pressure above 15 psi to improve coupling.

The wedge and lap shear test specimens prepared with the ultrasonic process (i.e., cured at 85 watts for 1 hour) had results which were comparable with the test results of the thermally cured specimens. DSC tests on the ultrasonically cured specimens also indicate a degree of cure comparable to the thermally cured specimens.

The ultrasonic energy needed to cure FM-73 depends on the bond area and the amount of aluminum substrate directly in contact with the bond area since aluminum is an excellent conductor of heat. Although ultrasonic energy is absorbed directly by the adhesive, aluminum acts like a heat sink and conducts away the heat generated in the adhesive. Therefore, the steady state temperature of the

adhesive drops if the aluminum substrate volume and surface areas increase. Therefore, it is necessary to compensate for the heat sink effect of aluminum in order to estimate the level of ultrasonic energy necessary to fully cure the adhesive.

In conclusion although some practical problems need to be solved, the feasibility of curing structural adhesive with ultrasonic energy has been demonstrated during this project.

Appendix

DSC Plots of the Adhesive Cured During the Box-Behnken Design Tests

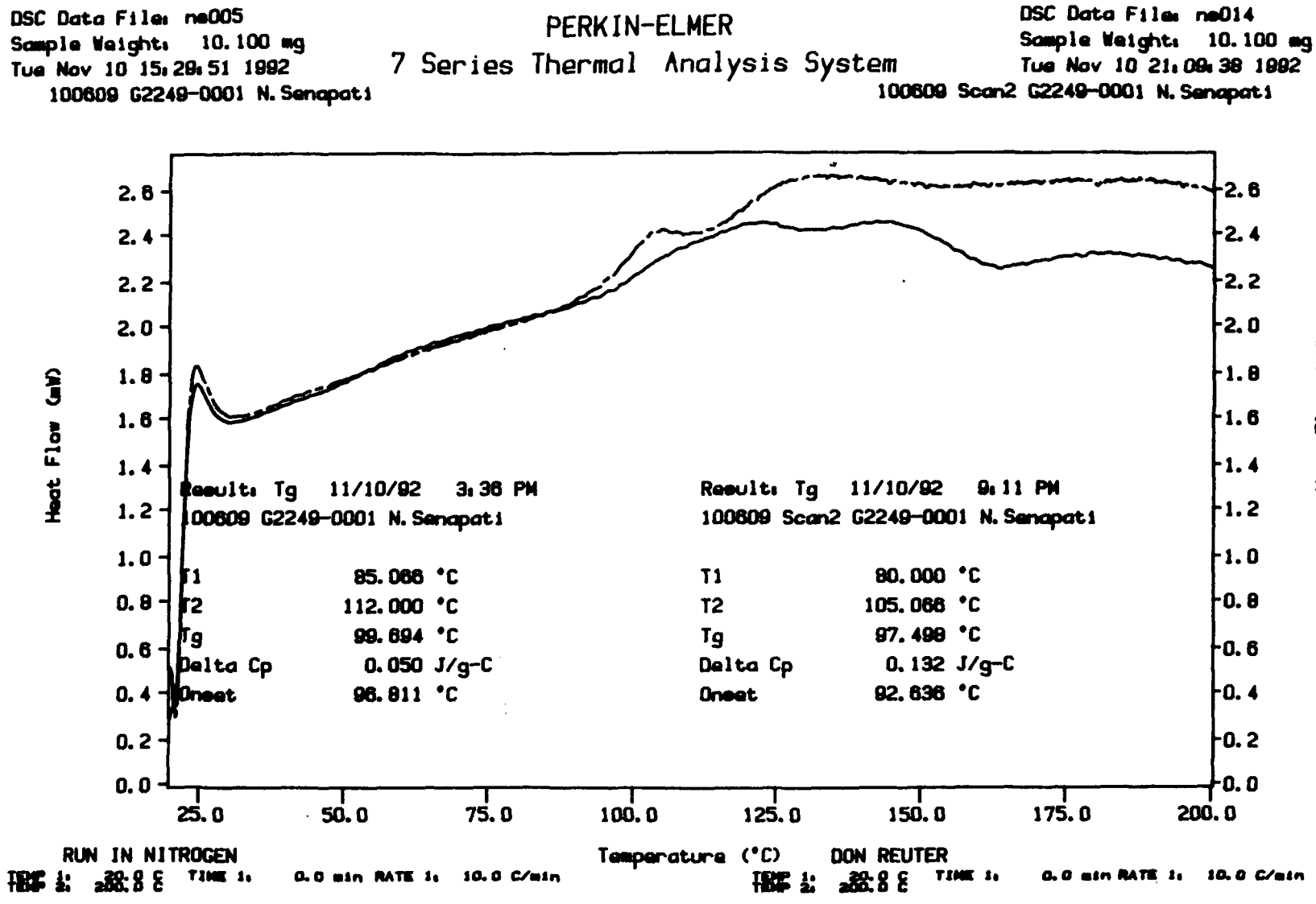

Figure 15. DSC plot of Specimen No. 100609.

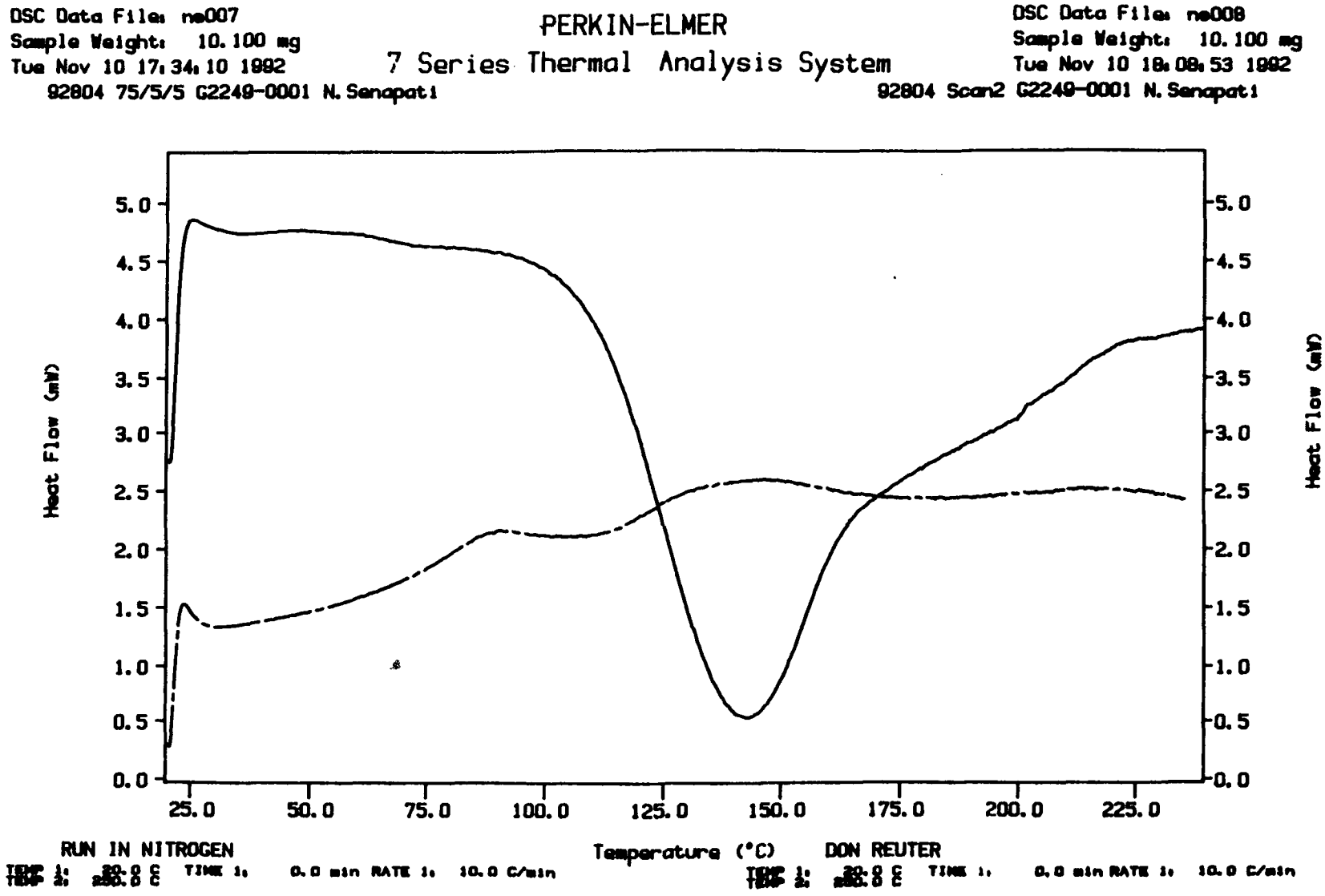

Figure 16. DSC plot of Specimen No. 92804.

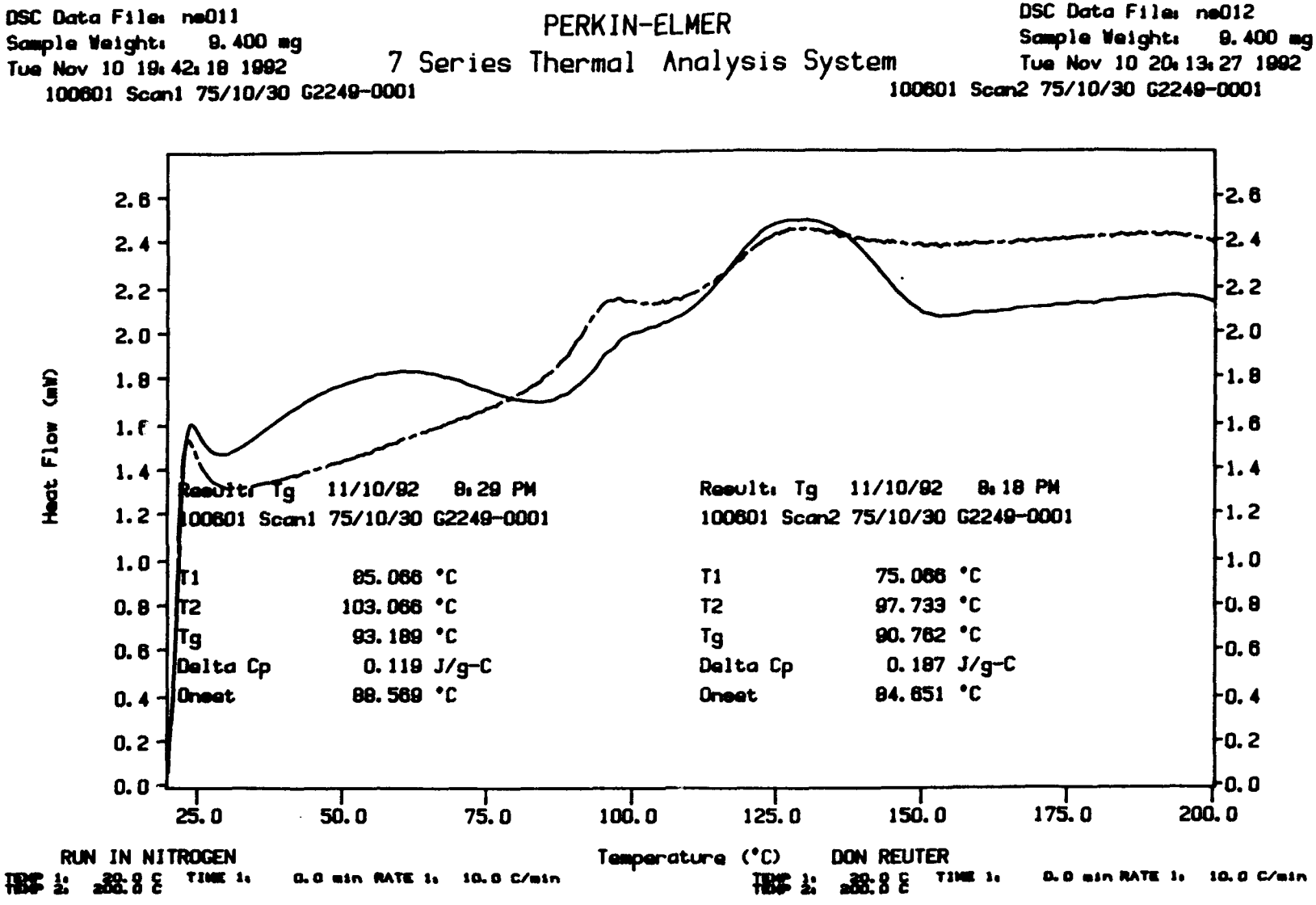

Figure 17. DSC plot of Specimen No. 100601.

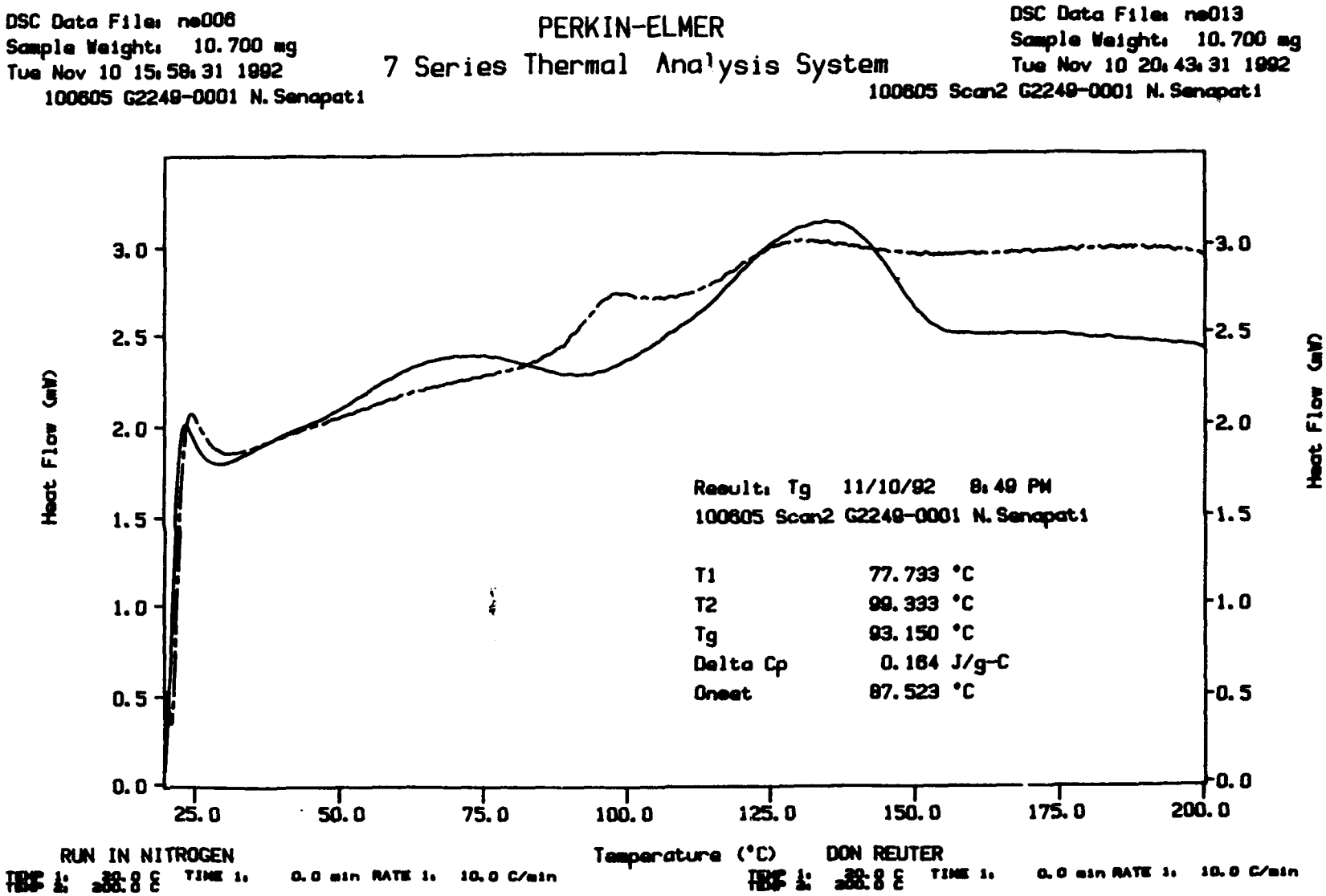

Figure 18. DSC plot of Specimen No. 100605.

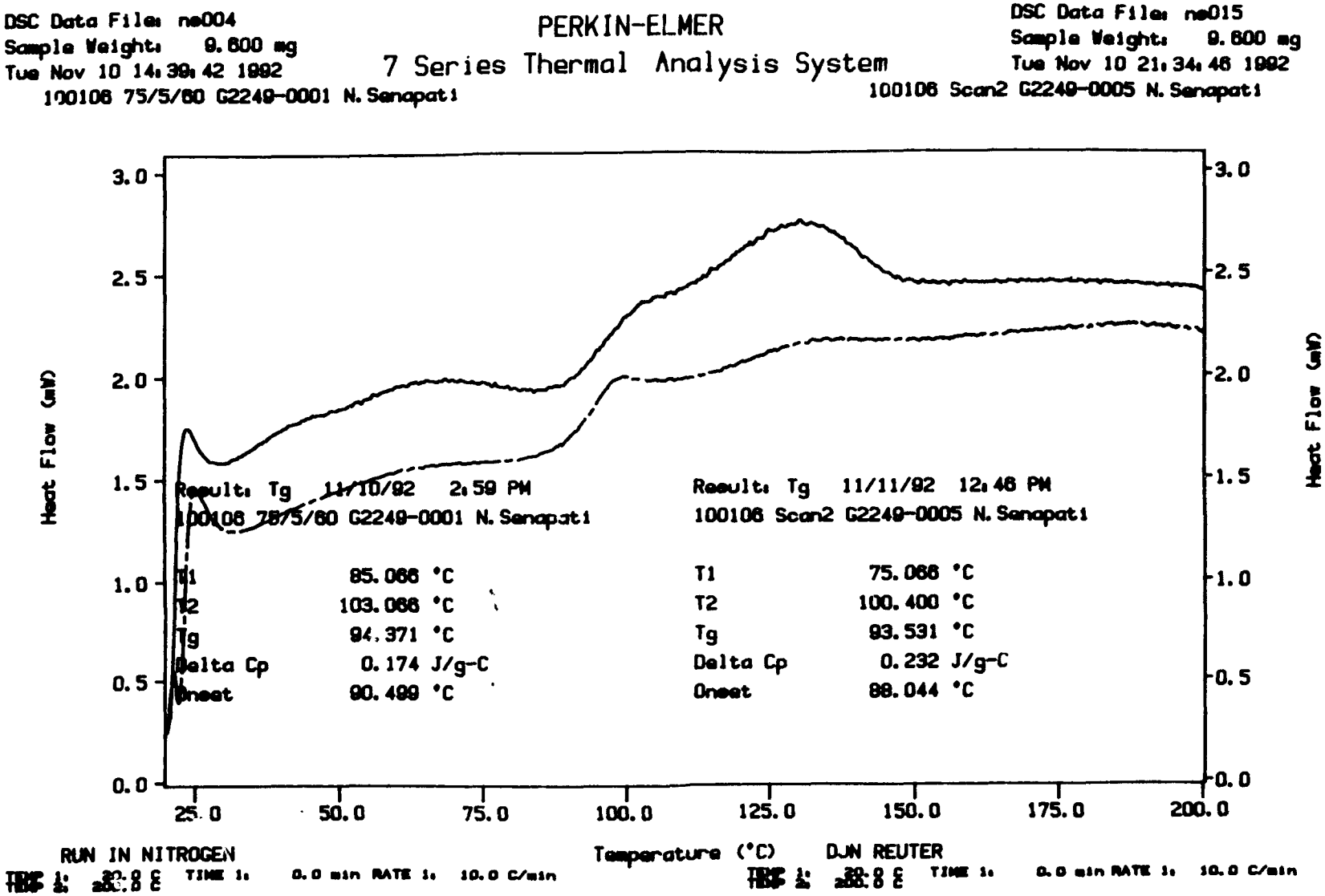

Figure 19. DSC plot of Specimen No. 100106.

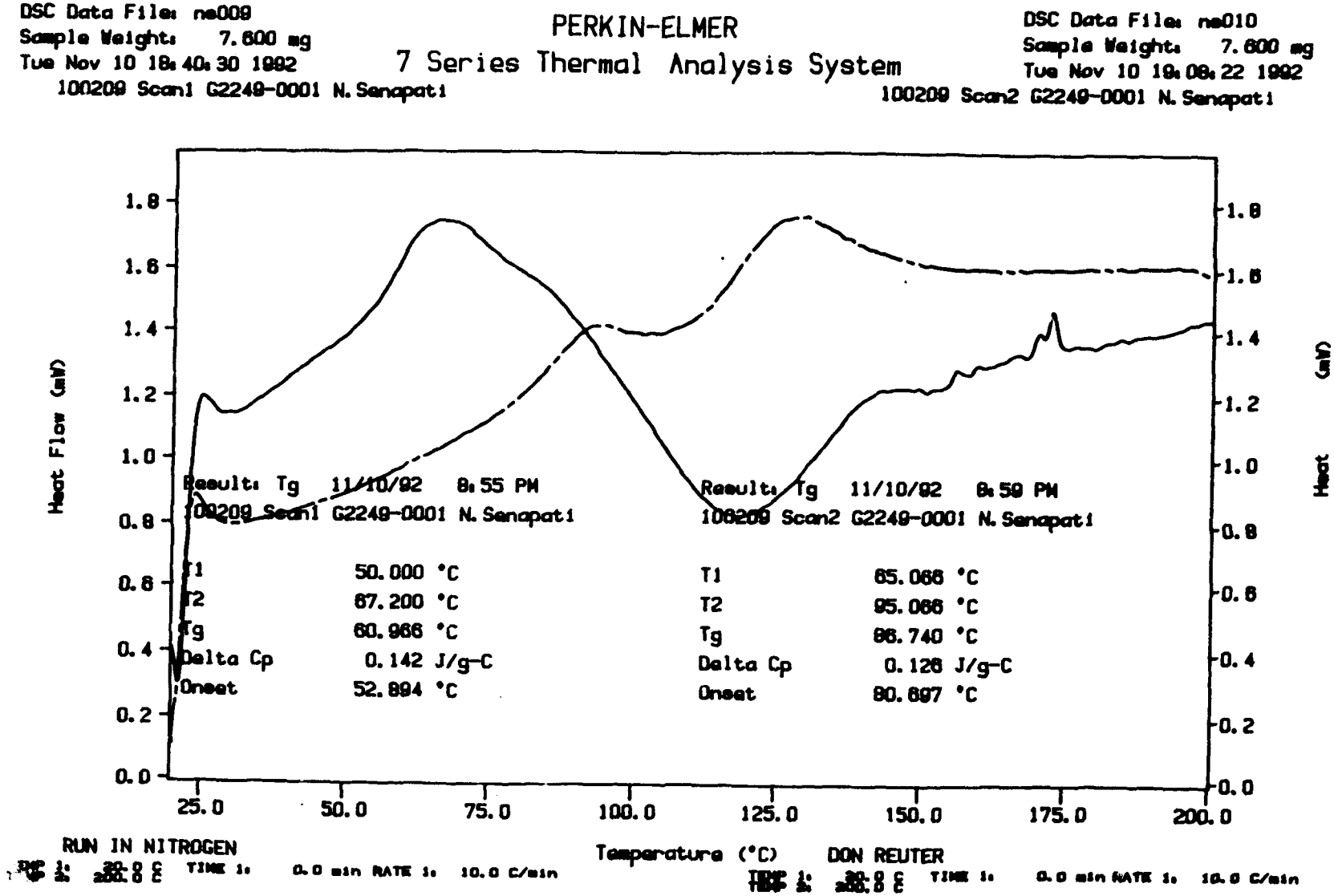

Figure 20. DSC plot of Specimen No. 100209.

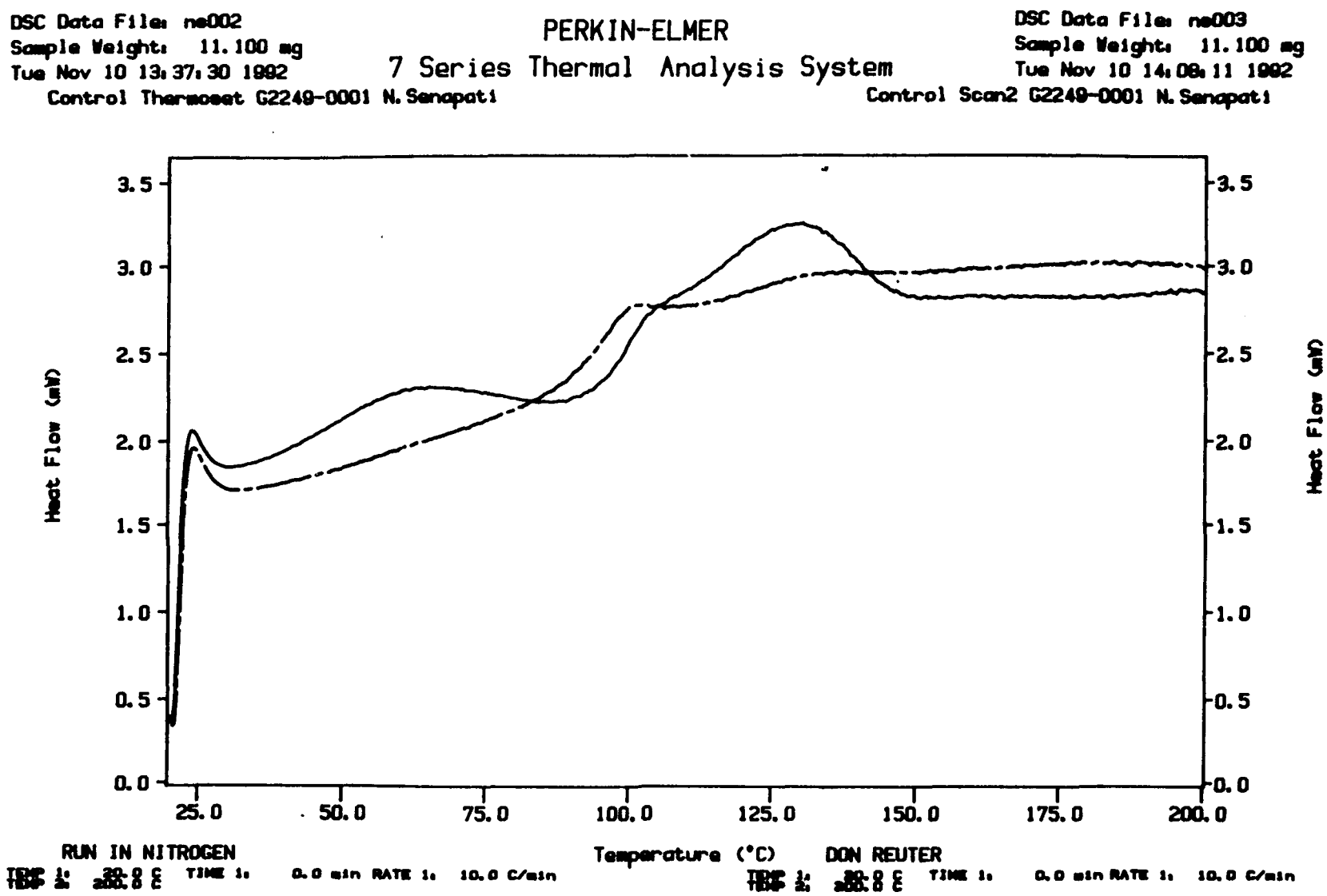

Figure 21. DSC plot of thermally cured specimen.